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PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of
Wilhelm et al.
Serial No. 09/821,876
Filed: March 30, 2001

Docket: 2000FR303
Group Art Unit: 1711
Examiner: S. Berman

For: Silico-Acrylic, Compositions Method for their Preparation and use.

DECLARATION UNDER 37 CFR § 1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Dear Sir:

I, Dr. Can Vu Ngoc, declare I am a citizen of France, residing in the city of Paris;

I am a chemist who has worked on the above-named Application;

I have a PhD in Chemistry from the University of Pierre et Marie Curie in France in 1977;

I was employed by Société Française Hoechst from 1977 to July 1997, and from July 1997 until the present I continued my employment with Clariant (France), as a

research Chemist,

I am well acquainted with the development and commercialization of organic polymers and fluid silico-acrylic compositions and have been specialized in this technology area since 2000. Since that time, and as part of my regular job duties, I have been responsible for the development of many fluid silico-acrylic compositions,

Applicants' invention is directed to an fluid silico-acrylic compositions, which are stable in the long term, polymerizable thermally or by radiation, by a mechanism giving rise to free radicals, and have a very low water and volatile solvents content, their preparation method and application of these to obtain abrasion and scratch- resistant coatings. The reference that the office bases its rejection on is Swofford US Patent 4,822,828, which teaches use of a multifunctional acrylates, where triacrylates are preferred and water-soluble triacrylates are more preferred.

Applicants' multifunctional alkoxylated (meth)acrylate monomers taught in the specification , namely SR 454 marketed by Cray Valley, and RTT-193 marketed by Servo Delden BV were used in the examples and are water insoluble.

The other multifunctional alkoxylated (meth)acrylate monomers taught in the specification, Ebecryl 1100 contains a typographical error, it should be Ebecryl 1160 an ethoxylated trimethylolpropane triacrylate marketed by UCB/Radcure which also should be water insoluble.

In the application the first 4 examples are all to compositions of the invention. Example 5 is the use of the compositions taught to produce a coating. Examples 1 and 2 of the instant invention used the alkoxylated (meth)acrylate monomers SR 454, Examples 3 and 4 of the instant invention used the alkoxylated (meth)acrylate

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monomers RTT-193.

EXAMPLE 1

The following are mixed under agitation in a reactor at ambient temperature:

- 122.4 g of an acid silica sol containing 40 wt.% silica, i.e. 49 g silica with an average diameter of 50 nm, and 60% water, i.e. 73.4 g water, and having a pH of 2.
- 396.4 g isopropanol
- 26 g vinyltrimethoxysilane, i.e. 0.53 g vinyltrimethoxysilane per gram initial dry silica.
- 125g of ethoxylated trimethylolpropane triacrylate with a molecular mass of 428.

The reactor is brought under reduced pressure of 50 to 110 mm of Hg and then gently heated in a double boiler so that the temperature of the boiler does not exceed 45°C and that of the reactional medium 35°C for 4 hours.

The water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.

After filtration, a slightly yellow, limpid, transparent solution, stable in the long term, is obtained containing 30 wt.% silica and 0.3 % water and having a Brookfield viscosity of 304 mPa.s determined at 20°C (speed 100 tr/mn; mobile no. 2).

It is noted that under appropriate storage conditions, i.e. in a cool, dark place, at the end of 6 months following the preparation of the product, there is still a clear, limpid, homogenous, transparent, slightly yellow solution, which is stable in the long term, and no phenomenon of turbidity is therefore found.

In the same way, the coatings obtained with this composition have no film.

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EXAMPLE 2

The following are mixed under agitation in a reactor at ambient temperature:

- 192 g of an acid silica sol containing 30 wt.% silica, i.e. 57.6 g silica with an average diameter of 50 nm, and 70 % water, i.e. 134g water and having a pH of 2.
- 971 g isopropanol
- 6 g vinyltrimethoxylsilane, i.e. 0.1 g vinyltrimethoxysilane per gram of initial dry silica
- 136.4 g ethoxylated trimethylolpropane triacrylate.

Under conditions analogous to those described in example 1, after 6 hours of reaction at 35°C, the water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.

A clear, limpid, transparent, slightly yellow solution which is stable in the long term is obtained, containing 29 wt.% silica and 0.4 wt.% water, and having a Brookfield viscosity of 180 mPa.s determined at 20°C (speed 100tr/mn; mobile no.2).

It is noted that under appropriate storage conditions, i.e. in a cool, dark place, at the end of 6 months after the preparation of the product, there is still a clear, liquid, homogenous, transparent, slightly yellow solution which is stable in the long term. Thus no turbidity phenomenon is noted. In the same way, the coatings obtained with this composition have no film.

EXAMPLE 3

The following are mixed under agitation in a reactor at ambient temperature:

- 219g of an acid silica sol containing 40 wt.% silica, i.e. 87.6 g silica with an average diameter of 12 nm and 60% water, i.e. 131.4g water and having a pH of 2.
- 945 g isopropanol
- 6 g vinyltrimethoxylsilane, i.e. 0.07 g vinyltrimethoxysilane per gram of

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initial dry silica

- 206.4 g ethoxylated pentaerythritol triacrylate.

Under conditions analogous to those described in example 1, the water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.

A clear, limpid, transparent, slightly yellow solution which is stable in the long term is obtained, containing 29 wt.% silica and 0.6 wt.% water, and having a Brookfield viscosity of 980 mPa.s determined at 20°C (speed 100tr/mn; mobile no.2).

It is noted that under appropriate storage conditions, i.e. in a cool, dark place, at the end of 6 months after the preparation of the product, there is still a clear, liquid, homogenous, transparent, slightly yellow solution which is stable in the long term. Thus no turbidity phenomenon is noted. In the same way, the coatings obtained with this composition have no film.

EXAMPLE 4

The following are mixed under agitation in a reactor at ambient temperature:

- 200g of an acid silica sol containing 30 wt.% silica, i.e. 60 g silica with an average diameter of 12 nm and 70% water, i.e. 140 g water and having a pH of 2.
- 1040 g isopropanol
- 16 g vinyltrimethoxylsilane, i.e. 0.27 g vinyltrimethoxysilane per gram of initial dry silica
- 124 g ethoxylated pentaerythritol triacrylate.

Under conditions analogous to those described in example 1, after 8 hours of reaction the water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.

A slightly yellow, limpid, transparent solution which is stable in the long term is obtained, containing 32 wt.% silica and 0.6 wt.% water, and having a Brookfield viscosity of 1470 mPa.s (speed 100tr/mn; mobile no.4).

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It is noted that under appropriate storage conditions, i.e. in a cool, dark place, at the end of 6 months after the preparation of the product, there is still a clear, limpid, homogenous, transparent, slightly yellow solution which is stable in the long term. Thus no turbidity phenomenon is noted. In the same way, the coatings obtained with this composition have no film.

We have now have also run comparative examples with the water-soluble triacrylates preferred by Swofford. The first two comparative examples use the preferred triacrylate of Swofford, Sartomer C-9035 and another comparative example with a different water soluble, triacrylate.

COMPARATIVE EXAMPLE 1

The following are mixed under agitation in a reactor at ambient temperature:

- 219 g of an acid silica sol containing 40 wt.% silica, i.e. 87.6 g silica with an average diameter of 12 nm and 60 % water, i.e. 131.4 g water and having a pH of 2
- 945 g isopropanol
- 6 g vinyltrimethoxysilane, i.e. 0.07 g vinyltrimethoxysilane per gram of initial dry silica
- 206.4 g of ethoxylated (15) trimethylolpropane triacrylate (SR-9035 marketed by Sartomer similar to CN 435 marketed by Cray Valley).

The reactor is brought under reduced pressure of 50 to 110 mm of Hg and then gently heated in a double boiler so that the temperature of the boiler does not exceed 45°C and that of the reactional medium 35°C for 4 fours.

The water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.

After filtration, a slightly yellow gel is obtained.

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COMPARATIVE EXAMPLE 2

The following are mixed under agitation in a reactor at ambient temperature :

- 122.4 g of an acid silica sol containing 40 wt.% silica, i.e. 49 g silica with an average diameter of 25 nm and 60 % water, i.e. 73.4 g water and having a pH of 2
- 396.4 g isopropanol
- 26 g vinyltrimethoxysilane, i.e. 0.22 g vinyltrimethoxysilane per gram of initial dry silica
- 125 g of ethoxylated (15) trimethylolpropane triacrylate (SR-9035 marketed by Sartomer similar to CN 435 marketed by Cray Valley).

Under conditions analogous to those described in comparative example 1, after 6 hours of reaction at 35 °C, the water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.
A whitish gel is obtained.

COMPARATIVE EXAMPLE 3

The following are mixed under agitation in a reactor at ambient temperature :

- 122.4 g of an acid silica sol containing 40 wt.% silica, i.e. 49 g silica with an average diameter of 25 nm and 60 % water, i.e. 73.4 g water and having a pH of 2
- 396.4 g isopropanol
- 26 g vinyltrimethoxysilane, i.e. 0.22 g vinyltrimethoxysilane per gram of initial dry silica
- 125 g of ethoxylated (20) trimethylolpropane triacrylate (SR-415 marketed by Sartomer).

Under conditions analogous to those described in comparative example 1, after 6 hours of reaction at 35 °C, the water-isopropanol mixture is distilled so that the quantity of residual water is below 1%.

A slightly yellow solution is obtained but having a Brookfield viscosity of 2200 mPa.s determined at 20°C (speed 10 tr/mn ; mobile no.2).

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Comparative examples 1 and 2 are clearly not a composition which is stable in the long term. Example 3 while not a gel has a viscosity higher than taught by the instant application. The compositions according to the invention are especially fluid. Their Brookfield viscosity after manufacture, determined at 20°C with a 100 tr/mn Brookfield RVT device is low (below 1500 mPa.s), which makes it possible to use them as they are without subsequent dilution by solvents.

In light of Applicants' comparative examples and the experimental data on Applicants' composition, Applicants'respectfully request that the obvious rejection to USP 4,822,828 Swofford, be withdrawn and the claims allowed.

The under signed declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

12/12/03
Date

Can Vu Ngoc
Dr. Can Vu Ngoc
Clariant (France)

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